- 19. Japan Patent Office (JP)
- 12. Laid-open Patent Gazette (A)
- 11. Laid-open Patent Application Hei 4-136174
- 43. Date Published: May 11, 1992

| 51. | Int. Cl. ⁵ C 23 C 16/30 B 23 B 27/14 B 23 P 15/28 C 23 C 14/02 14/06 | ID No. A A | Office Control No. 8722-4K 7632-3C 8709-3C 9046-4K 9046-4K | |
|-----|--|------------------|---|--|
| | 16/02 | | 9046-4K 8722-4K | |

Examination Not Requested Yet Number of Claims: 1 (Total 4 pages)

- 54. Invention Title: Method for Manufacturing Surface Coated Ceramic Cutting Tool with Improved Adhesion of Hard Coating Layer
- 21. Application Number: Hei 2-258281
- 22. Application Date: September 27, 1990
- 72. Inventor: Toshiyuki Takano, Mitsubishi Metals Central Laboratory, 1-297 Kita Fukuro-cho, Omiya City, Saitama Prefecture
- 72. Inventor: Yuichiro Terao, Mitsubishi Metals Tokyo Works, 1-27-20 Nishi Shinagawa, Shinagawa-ku, Tokyo
- 71. Applicant: Mitsubishi Materials Corp., 1-6-1 Otemachi, Chiyoda-ku, Tokyo
- 74. Representative: Kazuo Tomita, Patent Agent, and one other

SPECIFICATION

1. TITLE OF INVENTION

Method for Manufacturing Surface Coated Ceramic Cutting Tool with Improved Adhesion of Hard Coating Layer

2. WHAT IS CLAIMED IS:

(1) A method for manufacturing a surface coated ceramic cutting tool with improved adhesion of its hard coating layer, wherein a matrix consisting of a silicon nitride based ceramic or sialon based ceramic with a ground surface is heated in a nitrogen-containing atmosphere and held at 1050~1400°C to perform a matrix surface modification heat treatment, after which a hard coating layer is vapor deposited and formed under ordinary conditions.

3. DETAILED EXPLANATION OF THE INVENTION Industrial Field of Application

The present invention pertains to a method for manufacturing a surface coated ceramic cutting tool with improved adhesion of its hard coating layer, vapor-deposited

JP 4-136174

and formed on the surface of a matrix consisting of silicon nitride (hereinafter indicated by Si₃N₄) based ceramic or sialon based ceramic, to the aforesaid matrix surface.

Prior Art

A conventional and known method for manufacturing a surface coated ceramic cutting tool is as described in Examined Patent Application Sho 63-48835, for example. The main component of the matrix is α -sialon, represented by the formula $M_X(Si,Al)_{12}(O,N)_{16}$ (where $0 \le x \le 2$, and M is at least one of Y, Mg, a rare earth element, Ca, Li, and Na), or similarly β-sialon, represented by the formula Si_{6-Z}Al_ZO_ZN₈₋ z (where $0 < z \le 4.3$), or Si₃N₄. The sialon based ceramic or Si₃N₄ based ceramic matrix includes 1~20% (in the following, % indicates weight%) of a glass phase consisting of at least one of the elements included in the aforesaid M and an oxide of Si and/or Al (hereinafter indicated as M-(Si,Al)-O-N compound) as a binding phase forming component, and in addition when necessary includes 1~35% of at least one of carbides and nitrides of Ti, Zr, Hf, Si, and Al, for example, and solid solutions of two or more of these as a dispersed phase forming component. After the matrix surface is ground, ordinary chemical vapor deposition (CVD) or physical vapor deposition (PVD) is used to vapor deposit and form a hard coating layer, with average layer thickness 0.5~10 µm. consisting of a single layer or two or more layers of, for example, carbides, nitrides, carbonitrides, and oxycarbonitrides of Ti and Si, and oxides and oxynitrides of Al. It is also known that these surface coated ceramic cutting tools are often used for cutting cast iron.

Problems the Invention Is to Solve

Meanwhile, in recent years there has been dramatic progress in FA and NC cutting tools. This has been accompanied by demands for longer cutting tool life. But the aforesaid conventional surface coated ceramic cutting tools do not have sufficient adhesion of the hard coating layer to the matrix surface, so they are liable to delaminate in actual use, and this delamination causes them to have a relatively short useful life.

Means for Solving the Problems

Therefore the present inventors focused on the aforesaid conventional surface coated ceramic cutting tools from the perspective described above, and performed research intended to improve adhesion of the hard coating layer to the matrix surface. As a result, they discovered that:

- (a) The cause of insufficient adhesion of the hard coating layer to the matrix surface in conventional surface coated ceramic cutting tools is the glass phase consisting of an M-(Si,Al)-O-N compound that is present in the matrix as a binding phase forming component. Adhesion between this glass phase and the hard coating layer is very bad. Essentially, adhesion of the hard coating layer is ensured by the sialon or Si₃N₄ that is the main component.
- (b) However, when manufacturing the aforesaid surface coated ceramic cutting tool, if a heat treatment is performed on the matrix, after grinding its surface and before vapor depositing and forming the hard coating layer, under the conditions of heating it in a nitrogen-containing atmosphere and holding it at 1050~1400°C, the amorphous glass phase present at the grain boundaries reacts with the nitrogen

atmosphere and crystallizes. Adhesion of the crystal phase crystallized in this manner to the hard coating layer is very high, so adhesion of the hard coating layer to the matrix surface is greatly improved, and delamination of the hard coating layer when cutting is dramatically suppressed, and excellent cutting performance can be achieved for a long time.

The research results indicated in the aforesaid (a) and (b) were achieved. Therefore, the present invention is one based upon the aforesaid research results. It is characterized as a method for manufacturing a surface coated ceramic cutting tool with improved adhesion of its hard coating layer, wherein before forming a hard coating layer using ordinary CVD or PVD on the surface of a matrix consisting of a sialon based ceramic or an Si₃N₄ based ceramic, the ground matrix surface is heated in a nitrogen-containing atmosphere and held at 1050~1400°C to perform a matrix surface modification heat treatment, the glass phase constituting the binding phase of the matrix surface part is crystallized, and the hard coating layer is vapor deposited and formed on the matrix surface in this state.

Furthermore, in this inventive method the heating temperature of the heat treatment is set at $1050\sim1400^{\circ}\text{C}$ because at temperatures below 1050°C crystallization of the glass phase is inadequate, and as a result the desired effect of improving adhesion with the hard coating layer is not obtained. On the other hand, if the temperature exceeds 1400°C it causes a breakdown reaction in the sialon or Si_3N_4 that is the main component of the matrix, and tool strength decreases.

Embodiments

Next, the inventive method shall be explained in greater detail through embodiments.

The starting powders used were $\alpha\textsc{-Si}_3N_4$ powder with average grain size $0.8~\mu m$, Al_2O_3 powder with average grain size $0.5~\mu m$, AlN powder with average grain size $1.2~\mu m$, Y_2O_3 powder with average grain size $1.2~\mu m$, ZrO_2 powder with average grain size $0.5~\mu m$, MgO powder with average grain size $0.5~\mu m$, and TiN powder with average grain size $1.5~\mu m$. These starting powders were combined in predetermined mixing ratios, wet-mixed for 36 hours in a ball mill, dried, and then pressed into green compacts. The green compacts sintered for two hours at a temperature of $1750^{\circ}C$ in an N_2 atmosphere at one atmosphere pressure, thus forming matrixes consisting of $\alpha\textsc{-sialon}$ based ceramic, $\beta\textsc{-sialon}$ based ceramic, and Si_3N_4 based ceramic with the compositions shown in Table 1. These matrixes were ground to adjust their dimensions, after which heat treatment was performed to modify the matrix surface under the conditions indicated in Table 1. Then hard coating layers with the compositions and layer thicknesses shown in Table 1 were formed on the matrix surfaces using CVD under ordinary conditions. Thus inventive methods $1{\sim}12$ manufactured surface coated ceramic cutting tools with the shape JIS SNGN120412.

Also, for comparison, conventional methods 1~3 were performed under the same conditions, except for not performing a heat treatment to modify the matrix surface, to manufacture surface coated ceramic cutting tools.

Next, dry continuous high-speed cutting tests on cast iron were performed using these various surface coated ceramic cutting tools under the following conditions:

Workpiece: Round bar of cast iron (FC25)

Cutting speed: 350 m/min

Feed: 0.2 mm/rev Depth of cut: 1.5 mm

The cutting time until the end of the tool's useful life was measured. These measurement results are shown in Table 1.

Effect of the Invention

The results shown in Table 1 clearly show that inventive methods 1~12 resulted in superior hard coating layer adhesion, so wear of the hard coating layer was reduced (normal wear), and it was possible to manufacture a surface coated ceramic cutting tool that could cut for a long time until the end of the tool's useful life. On the other hand, in the surface coated ceramic cutting tools manufactured by conventional methods 1~3 the glass phase consisting of an M-(Si,Al)-O-N compound that was present in the matrix as a binding phase forming component impaired adhesion of the hard coating layer to the matrix surface, so delamination of the hard coating layer occurred in a relatively short time, and caused the tool to end its useful life.

As described above, the inventive method makes it possible to manufacture a surface coated ceramic cutting tool with improved adhesion of the hard coating layer to the matrix surface, so when this is practiced one can achieve improved cutting performance for a long time, and it is suitable for FA and NC cutting machines, so it has effects that are advantageous in industry.

Applicant: Mitsubishi Metals Corp.

Representative: Kazuo Tomita, Patent Agent, and one other

| Инск. Сотр. Инск. cutting (µm) (µm) time (m) 6 TiN 0.5 15 6 TiN 0.5 17 | Comp. Thick. (µm) TiN 0.5 TiN 0.5 TiN 0.5 TiN 0.5 TiN 0.5 | Comp. I hick. (µm) TiN 0.5 TiN 0.5 TiN 0.5 TiN 0.5 TiN 0.5 | Comp. Thick. (µm) TiN 0.5 TiN 0.5 TiN 0.5 TiN 0.5 TiN 0.5 TiN 0.5 Al ₂ O ₃ 2 Al ₂ O ₃ 2 | Comp. I hick. (µm) TiN 0.5 TiN 0.5 TiN 0.5 TiN 0.5 TiN 0.5 Al ₂ O ₃ 2 Al ₂ O ₃ 2 Al ₂ O ₃ 2 | Comp. I hick. (µm) TiN 0.5 TiN 0.5 TiN 0.5 TiN 0.5 Al ₂ O ₃ 2 Al ₂ O ₃ 2 Al ₂ O ₃ 2 | Comp. Thick. (µm) TiN 0.5 TiN 0.5 TiN 0.5 TiN 0.5 Al ₂ O ₃ 2 | Comp. Thick. (µm) TiN 0.5 TiN 0.5 TiN 0.5 TiN 0.5 Al ₂ O ₃ 2 Al ₂ O ₃ 7 TiN 0.5 | Comp. Thick. (µm) TiN 0.5 TiN 0.5 TiN 0.5 TiN 0.5 Al ₂ O ₃ 2 TiN 0.5 TiN 0.5 TiN 0.5 TiN 0.5 |
|--|---|---|---|--|--|---|---|---|
| NIT NIT | | | Tin Tin Tin Tin Tin Al ₂ O ₃ | Tin Tin Tin Tin Al ₂ O ₃ Al ₂ O ₃ | TiN TiN TiN TiN TiN Al ₂ O ₃ Al ₂ O ₃ | TIN | TiN TiN TiN TiN Al ₂ O ₃ Al ₂ O ₃ Al ₂ O ₃ | TIN |
| | | | | | | | | |
| | Sic 2 TiC 6 | | | | | | | |
| 5 | I SIC | 3 - L | | | | | | |
| | Tich | | | | | | | |
| 3 | 0.5 | 5 - 1 | 3 3 0.5 | | | | | |
| | 1200 | 1200 1400 1050 1200 | 1200 1400 1050 1200 1200 1400 | 1200 1400 1050 1200 1200 1400 | 1200 1400 1050 1200 1400 11050 | 1200 1400 1050 1200 1400 11200 11200 | 1200 1400 1050 1200 1200 1200 1200 1200 | 1200 1400 1200 1200 1200 1200 1200 1200 |
| | 0.5 atmo. N ₂ 0.5 atmo. N ₂ | 0.5 atmo. N ₂ 0.5 atmo. N ₂ 1 atmo. N ₂ 1 atmo. (N ₂ + 1 oth) | 0.5 atmo. N ₂ 1 atmo. N ₂ 1 atmo. (N ₂ + 10% H ₂) 0.5 atmo. N ₂ 0.5 atmo. N ₂ | 0.5 atmo. N ₂ 1 atmo. N ₂ 1 atmo. N ₂ 1 atmo. (N ₂ + 10% H ₂) 0.5 atmo. N ₂ | 0.5 atmo. N ₂ 1 atmo. N ₂ 1 atmo. (N ₂ + 10% H ₂) 0.5 atmo. N ₂ 1 atmo. (N ₂ + 10% H ₂) 1 atmo. (N ₂ + 1 atmo. (N ₂ + 1 atmo. (N ₂ + 20% Ar) | 0.5 atmo. N ₂ 1 atmo. N ₂ 1 atmo. (N ₂ + 10% H ₂) 0.5 atmo. N ₂ 1.5 atmo. N ₂ 1.1 atmo. (N ₂ + 1.2 atmo. N ₂ 1.3 atmo. (N ₂ + 50% Ar) 1 atmo. (N ₂ + 50% Ar) | 0.5 atmo. N ₂ 1 atmo. N ₂ 1 atmo. (N ₂ + 10% H ₂) 0.5 atmo. N ₂ 0.5 atmo. (N ₂ + 1 atmo. (N ₂ + 1 atmo. (N ₂ + 1 atmo. (N ₂ + 50% Ar) 1 atmo. (N ₂ + 50% Ar) 0.5 atmo. N ₂ | 0.5 atmo. N ₂ 1 atmo. N ₂ 1 atmo. (N ₂ + 10% H ₂) 0.5 atmo. N ₂ 0.5 atmo. (N ₂ + 1 atmo. (N ₂ + 1 atmo. (N ₂ + 1 atmo. (N ₂ + 50% Ar) 1 atmo. (N ₂ + 50% Ar) 0.5 atmo. N ₂ 0.5 atmo. N ₂ |
| 1. | | β-sialon: remainder | <u>_ l_ l l_ l l</u> | | | <u></u> | <u></u> | _ |
| | | ZrO ₂ : [| | | | | | |
| | _ = | Y-Si-Al-O-N 2 compound: | z | z | i-Al-O-N pound: I-O-N | z | i-Al-O-N pound: I-O-N | i-Al-O-N pound: I-O-N |
|] | 1 4 3 | | | | | | | |
| | 1 | | d ve | 1 | 1. 6 1. 6 | 1 | ± | Convent. Method Inventive Method Convent. Method Inventive Method |

JP 4-136174

Table 1